Influence of Varying Rockwool Fibre Length on Properties of Intumescent Coating

by

See Yun Chuan

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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(AP Dr. Puteri Sri Melor Megat Yusoff)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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(SEE YUN CHUAN)

ABSTRACT

Intumescent coating is the most common passive fire protection for steel structure. Four formulations of intumescent coating reinforced with different Rockwool fibre lengths, i.e. 4 mm, 6 mm, 8 mm and 10 mm, and a controlled formulation, were developed and tested accordingly to investigate the influence of variable lengths on the properties of the intumescent coating and its char.

In the 800^{o}C furnace fire test, intumescent coatings expanded 6 to 10 times their initial thickness and experienced weight loss between 72-79%. The formulation with longest fibre expanded and lost weight the least and appeared to be more compact, denser and contained smaller pores. In the direct fire test of ~1022^{o}C, formulation with longer fibre retarded flammability better, enhanced the adhesion of the coating to the steel substrate and the ability to retain its original form, and provided thermal insulation up to 85-88% of directed heat. From the char strength test, it was found that the formulation with the longest fibre has the highest strength to sustain the char structure before destruction.

Degradation temperature, T_d, was determined to be at ~380^{o}C for all formulations in TGA yet residual weight increases with the increase in fibre lengths. From the XRD test, char samples of all formulations appeared to be in amorphous state and the major components determined were boron phosphate and boric acid.

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ABBREVIATIONS AND NOMENCLATURES

APP	Ammonium Polyphosphate
ATH	Alumina Trihydrate
DFT	Dry Film Thickness
MEL	Melamine
PER	Pentaerythritol
SSA	Sodium Sulphate Anhydrous
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction

CHAPTER 1
INTRODUCTION

1.1 Background Study

Structural steel is a common material used in construction, offshore, military and other industries. Structural steel exhibits good ductility but loses its structural properties when the temperature exceeds 500-600^{o}C [1-2]. Passive protection of structural steel members against fire in buildings, construction industry has become a very important issue. It is the prime requirement of building regulations of most countries to prevent the structural collapse of building to ensure safe evacuation of personnel in the event of fire, save precious human lives and assets. Passive fire protections are insulating systems designed to decrease heat transfer from the fire to the structure being protected, such as panels and blankets [3].

The use of intumescent fire retardant coating is one of the oldest, easiest, most economical and most efficient way to protect substrate from fire. It prevents heat from penetrating and flames from spreading by swelling upon heating and form char, which acts as a heat transfer barrier, as shown in Figure 1.1 [4-6]. Besides, it does not change the intrinsic properties of the substrate material and is easily processed. Other than steel or metallic materials, it can also be used on textiles, wood and polymers.

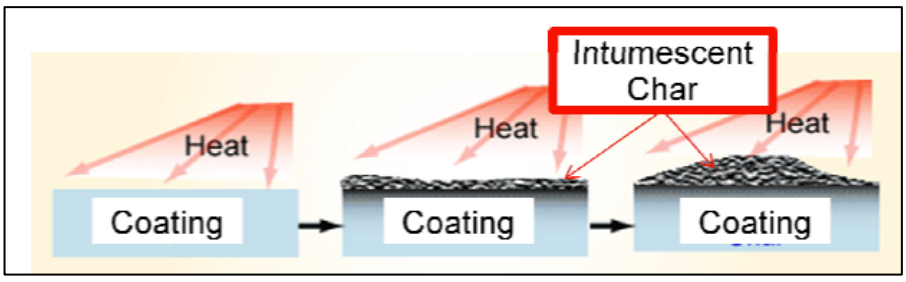


Figure 1.1: Swelling of intumescent coating to char formation.

The intumescent coating that is formulated for this study contains three main ingredients – ammonium polyphosphate (APP) as acid source, pentaerythritol (PER) as carbon source, and melamine (MEL) as blowing agent. Boric acid is also one of the main additive in the formulation for this study. This intumescent coating is epoxy resin based reinforced by mineral fibre, taken from Rockwool. Rockwool are made from long fine fibres spun from natural rock bonded with a thermosetting resin. It is originally intended as an insulation material suitable for hot surfaces up to 650°C or more and come in the form of blankets, slabs and pipe sectionals.

1.2 Problem Statement

The process of intumescence is complex and remains poorly understood despite the fact that it has been exploited commercially. The formulation of the coatings has to be optimized in terms of physical (char strength, expansion etc.) and chemical (thermal stability, reactivity etc.) properties so that an effective char can be formed. Since the char must be highly porous to provide thermal insulation, the relative thickness of the solid matrix must be low, causing difficulties in maintaining sufficient strength for the char to remain in place during a fire. Besides, the char may be consumed by physical erosion, oxidation of air, or attack of free radicals during the fire, causing it to degrade, loses its strength and fall off or blown away [6]. To counter the problem of poor char strength and adhesion, fibre sheets or mesh is applied onto the substrate prior to coating. When pressed into sheets, their ability to partition air makes it a good heat insulator [7-8]. However, they still poses certain disadvantages, such as difficulty of application, longer curing time, and thicker coating. Discontinuous fibres are used to overcome these problems by replacing the impregnation of fibre sheets to matrix. Discontinuous fibre reinforced intumescent coating can display similar strength and thermal properties compared to fibre sheets lay up, yet it is relatively new in the art where not much research has been done to further quantify the advantages of discontinuous fibres over fibre fabrics. It is yet to be understood about the influence of the fibre length on the coating properties such as the microstructure, mechanical strength and thermal insulation performance. Thus, this study will pose a respectively new contribution to the field of discontinuous fibre reinforced intumescent coating.

1.3 Objective/Scope of Study

Various tests were done to examine the influence of varying Rockwool fibre length on the properties of the intumescent coating. Furnace fire test and direct fire test were carried out to examine the difference in char formation. In direct fire test, the insulation performance was determined by attaching thermocouple beneath the steel substrate. Qualitative and quantitative observations were done to the physical properties of the char samples from both fire test. TGA was done to investigate the changes in weight with respect to temperature, while XRD was carried out to inspect the chemical compositions and crystal structure of the char. Strength test was carried out to find out the ability of the char to withstand crushing force.

CHAPTER 2
LITERATURE REVIEW/THEORY

2.1 Theory

Intumescence is the swelling of substances when heated. Intumescent coatings expand on high ratios in response to heat to form highly porous char – multicellular or foamed cellular layers that is thermally insulating, delays heat transfer and rapid increase of temperature to the underlying material in the event of fire [5-6]. Thus this implies greater time available for fire fighting and evacuation, provided that the expanded char stays in place during the fire. For steel, intumescent coatings are designed to perform under severe conditions and to maintain the steel integrity between 1 to 3 hours when the surrounding temperature is in excess of 1100^{o}C. The coatings are usually applied at a dry film thickness of few mm and do not interfere with the architectural aesthetics of the steel member.

Intumescent coatings contain three main ingredients – an acid source (usually ammonium polyphosphate/APP), a carbon source (such as pentaerythritol/PER), and a blowing agent (such as melamine/MEL), all bound together by a binder, either organic, inorganic or hybrid. When the coating surface reaches a critical temperature, the surface begins to melt and converted in highly viscous liquid, i.e. the acid source breaks down to form a mineral acid that takes part in the dehydration of the carbon source to form char. The blowing agent then decomposes to yield inert gaseous products with low thermal conductivity that trap inside the viscous fluid, causing char to expand, foam or swell [9-13]. Meanwhile, the binder contributes to the formation of uniform foam structure and char layer expansion. Usually epoxy resin is used for steel protection against hydrocarbon fire and jet fire (reaches ~1000^{o}C within several minutes), and is also very popular even in the aviation or aerospace industry [9]. The mechanism of intumescent coating in the case of fire and its

foaming structure are being shown in Figure 2.1 and Figure 2.2. The basic properties of the main ingredients used in this study are presented in Table 2.1.

Fibre reinforcement to the intumescent coating improves the strength, mechanical properties and thermal insulation performance of the char. In this literature, the term "discontinuous fibres" is used to differentiate from fibre fabric or woven fibre that is usually used in textiles. Discontinuous fibres are hereby defined as randomly aligned short fibres of length shorter than 10 mm.

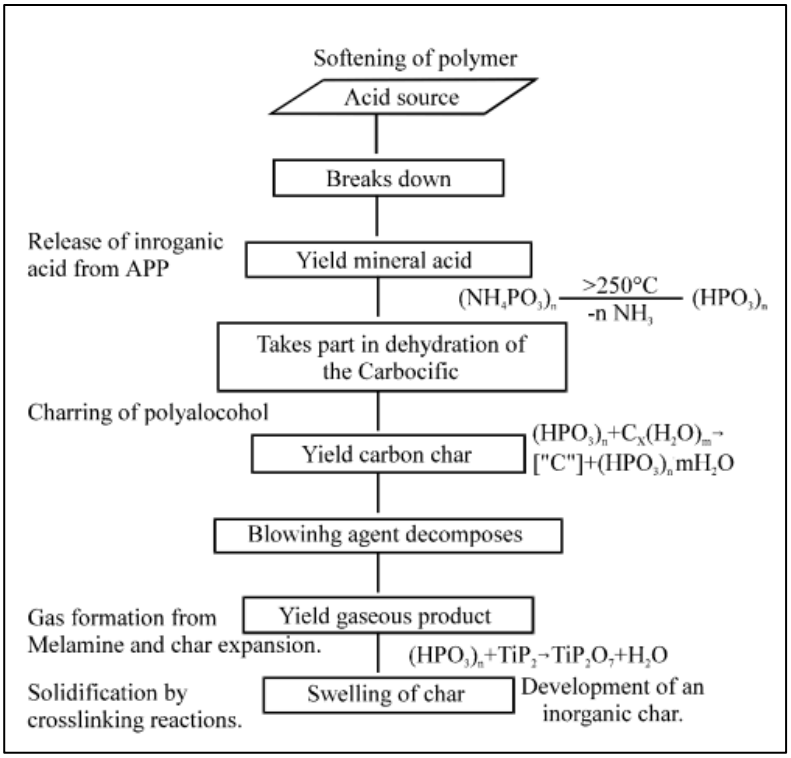


Figure 2.1: Mechanism of intumescent coating in the case of fire [2].

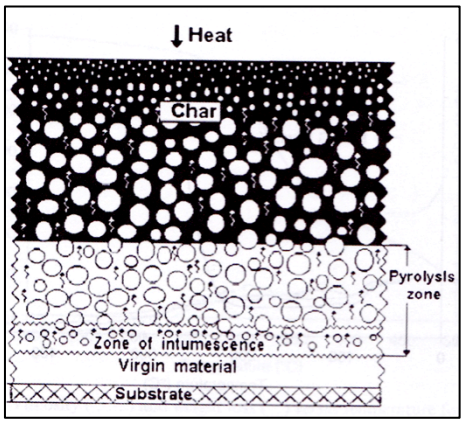


Figure 2.2: Foaming of the intumescent structure [13].

Table 2.1 Properties of the main ingredients used in intumescent coating formulation [14].

Ingredient	Properties
Ammonium Polyphosphate (APP), $(NH_4PO_3)_n$	Acid source; good flame retardant properties.
Pentaerythritol (PER), $C_5H_{12}O_4$	Carbon source; excellent water and alkali resistance and good aging characteristics.
Melamine (MEL)	Blowing agent; low smoke density and toxicity, excellent flame retardant properties, retard ignition by causing heat sink through endothermic dissociation.
Boric Acid, H_3BO_3	Additive; good flame retardant properties, provide excellent intumescent effect when mixed with APP in epoxy resin.

2.2 Literature Review

The behaviour of intumescent coating in terms of fire remains poorly understood and usually can only be investigated through fire tests. Due to the effect of different heat regimes and materials classification, various standard furnace fire tests or industrial tests has been done to investigate the particular protected construction in full scale. However to minimize the requirements of extensive efforts and costs, studies have been done to develop smaller scale analyses by means of laboratory test that could help predict the behaviour of the coating which then correlated to industrial furnace test or in the event of real fire [15]. The use of cone calorimeter in bench scale tests was found comparable with large-scale test up to a certain extent and rheometer was proven to be a powerful tool to investigate the mechanical resistance and expansion of the coating [16-17]. Kinetics analysis and mathematical modeling have been constantly developed in line with the small-scale tests to predict the thermal behaviour of intumescent coatings [18-19].

The uniqueness of each intumescent coating formulation added into the complexity of its mechanism and thus the interaction of binders and fillers were often studied.

To relate to a good protection of heat insulation, a certain extent of the swell formation and excessive cavities within charring layers are necessary. Studies have found that if the action by blowing agent occurs too early, the intumescent action is reduced because the material is too viscous; whereas if the action occurs too late, solidification of char inhibits the intumescence process. If the material becomes too fluid and has very low viscosity, large cells are formed during the blowing process. The char becomes frangible and ineffective as insulators. Thus the insulation efficiency of char depends on the cell structure, which in turn relates to the reaction of binders and fillers during the blowing process [20-21].

Meanwhile, the compactness of char provides mechanical resistance and strength. In most studies, a strong intumescent effect occurs under the mixture of APP and boric acid with the presence of epoxy resin. Studies have concluded that individually, boric acid and APP behaves like intumescent but it is too light and exhibits poor mechanical resistance. The best result is only obtained when both additives are combined into epoxy resin, where the char exhibits both high expansion and appropriate mechanical resistance [20, 22]. It is also agreed other studies, where a higher weight percentage of APP and boric acid when added into epoxy resin, the coating exhibits high heat stability as the charring layer is compact and has excessive cavities. It also exhibits good adhesion, where steady charring layer is formed and coating is prevented from falling off [23-24]. The compactness of coating was further improved by other studies that combined epoxy and sodium sulphate anhydrous (SSA), which increases the crosslinking degree of polymer binder. The interaction of fire retardant additives and the mixed binder has led to the formation of a better foam structure of char layer, increased intumescent rate and residue weights of coating [25].

For a char to be excellent for overall fire protection, it needs to have good heat shielding properties and at the same time not easily destructible by mechanical action. This in turn depends on the char cap height and its structure. The processes of char mechanical destruction have been analyzed for various types of chars in hot and cold states using various methods to study the mechanical strength. has also been studied. The rheological approach to destruction is the most common method found in the literature [26-27].

Varieties of intumescent coating are used for different markets and applications. Epoxy based coatings are used and favoured when high strength lightweight ratio, exterior durability, good mechanical properties and corrosion resistance are required. Besides high temperature, it also has good adhesion between char and adjacent substrate well below room temperature and normal service temperature, causing it to be favourably used in the petrochemical industries [28-30]. Adhesion properties of coating depend on the shrinkage of the coating after curing. The shrinkage introduces residual stress within the coating, which if exceeds the adhesion force, will cause the coating to fall off from the protected substrates. Epoxies tend to have lower volume reduction compared to other chemistries when cured, thus lower shrinkage, and in turn displays better adhesion properties [30].

To further enhance the properties of char forming coating, inclusion of fibres as reinforcement has been investigated. Fibres have lately been used as reinforcement materials in composite structures to support structural strength and enhance mechanical properties. It serves as many other purposes such as lightweight, insulative, and anti-corrosion. Fibres can also exhibit fire retardant properties as it also form chars, yet the char becomes oxidized and brittle when come to higher heat (>400^{o}C). Studies thus included dispersion of intumescent formulation onto fibre fabrics. When heated, the fibres interact with the intumescent and form char bonded structure, which enhances their flame and heat resistant properties [31-33]. This char-bonded structure increases mechanical stability and the expanded char that acts as an insulator offers good heat resistant properties to underlying substrates.

These have enabled textile composite materials to develop unusually high levels of flame and heat resistance. Many composite materials are layered, and each layer of fibre reinforcement acts as an insulator that affects the burning of the underlying resin. The heat that reaches the surface causes degradation of the resin leading to its ignition and migrates all the way down until all layers of the resins are burnt. Fabrics based on only inorganic fibres (glass, silica or alumina) were found very good protective flame and heat barriers that can withstand temperatures up to 1100°C for a considerable time [33]. When these woven fibre fabrics are sandwiched or impregnated onto epoxy resin, their thermal resistance properties further enhanced.

Impregnating fibre fabrics onto resins requires high cost, longer curing time, difficulty in application, and result in thicker coating. Studies have then incorporated discontinuous fibres into coating formulation to replace the impregnation or lay up of fibre fabrics.. Commonly used fibres include organic fibres such as carbon fibre, and inorganic fibres such as ceramic fibres, glass or other mineral fibres. Discontinuous fibres reinforced material exhibits good handling properties and can be cured to a hard and durable coating. The presence of discontinuous fibres in epoxy resin based intumescent coating has confirmed to reinforce the residual char by having compact yet porous char layer that consequently improved the fire-resistant properties. The char bonded structure formed promotes more char volume, better compactness, stronger structure and higher resistance to fire [34-36].

As with many aspects of the field in intumescent coating field, no prior published research is available thus far on the influence of varying discontinuous fibre length to the mechanical properties, structure and thermal insulation of char. Hence it is an area well to be explored.

CHAPTER 3
METHODOLOGY

The project was carried out in **three** stages: (i) material preparation, (ii) material testing, and (iii) material characterization. The description of each stage was discussed as below.

3.1 Material Preparation

3.1.1 Steel Substrate Preparation

A large sheet of structural steel (thickness: 25 mm) was polished with sand paper, primer coated with Dulux Epoxy-Zinc Phosphate (thickness: 2 mm), and cut into **twelve** 50 mm x 50 mm and **forteen** 100 mm x 100 mm sheets, as shown in Figure 3.1. Three sets of samples were formulated for each size of sheets. 50 mm x 50 mm steel sheets were designated as RA1, RA2, RA3, and RA4 for each set. The same goes for the 100 mm x 100 mm steel sheets, only that the designation of RB1, RB2, RB3 and RB4 were used. An extra sheet was used for the controlled formulation and another was intentionally left uncoated.

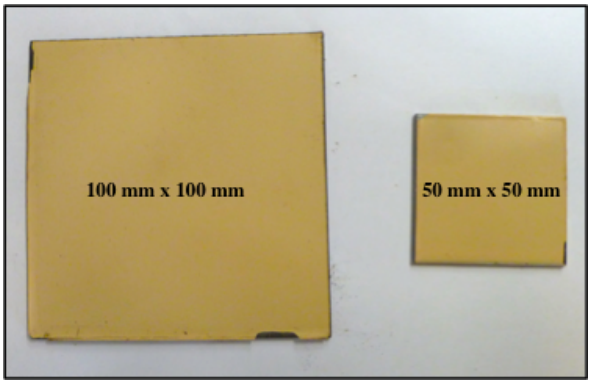


Figure 3.1: Primer coated steel sheets.

3.1.2 Intumescent Coating Formulation

Ammonium polyphosphate (APP), pentaerythritol (PER), melamine (MEL), boric

acid (H_3BO_3), talc, clay, fumed SiO_2 were mixed and grinded using Rocklab grinder as shown in Figure 3.2. Mineral fibres obtained from Rockwool was cut to varying length of 4 mm, 6 mm, 8 mm, and 10 mm, and mixed together with the other ingredients using Camfro mixer as shown in Figure 3.3.

Figure 3.2: Rocklabs grinder.

Figure 3.3: Camfro mixer.

Epoxy liquid and hardener (modified amide) were then added to the blend using Camfro automatic mixer with an average speed of 20 rpm for 3 minutes.

Figure 3.4: Intumescent ingredient mixture.

The weight composition of the ingredients is shown in Table 3.1. In total, **four** different formulations of **50 g** designated as formulation R1, R2, R3 and R4 were prepared using different fibre length of Rockwool fibre, 4 mm, 6 mm, 8 mm, and 10 mm, respectively. An extra formulation is prepared without including fibres to serve as a controlled sample, designated as RR. Each formulation was evenly applied onto the steel sheets respectively with spatula. A 50 mm x 50 mm steel sheet was coated with approximately 12.5 g of coating while a 100 mm x 100

mm steel sheet was coated with 50 g of coating. Before the coating was completely cured, it was trimmed manually along the edge of the steel sheet. Figure 3.5 shows the cured and trimmed intumescent-coated steel sheets. The coating was allowed to dry and cure naturally for 1 week in room temperature. The weight and final dry film thickness of coating were recorded using weighing machine and digital thickness scale machine.

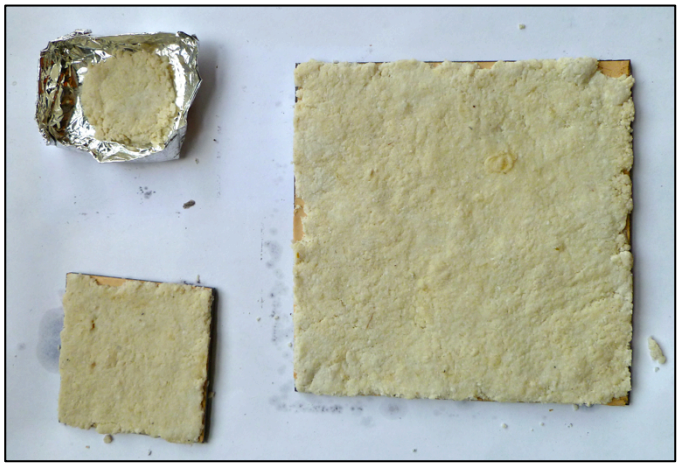


Figure 3.5: Intumescent-coated steel sheets.

Table 3.1: Weight composition of each ingredient in the intumescent formulation.

Ingredient	**Weight Percentage**
Ammonium polyphosphate (APP)	13.87 %
Pentaerythritol (PER)	4.62 %
Melamine (MEL)	4.62 %
Boric Acid	6.31 %
Talc	8.44 %
Clay	2.14 %
Fumed Silica Dioxide (SiO_2)	1.05 %
Epoxy	34.82 %
Hardener	17.41 %
Rockwool mineral fibre (4 different lengths)	0.42 %
Total	**100.00 %**

3.2 Material Testing

3.2.1 Furnace Fire Test

Each set of intumescent-coated steel substrates (50 mm x 50 mm) was fired in the electric furnace (Figure 3.6) from 25°C to 800°C in 20 minutes and dwelled for 60 minutes. The char samples were then allowed to cool down naturally in room temperature before further tests were carried out.

Figure 3.6: Electric furnace.

3.2.2 Direct Fire Test

Intumescent-coated steel substrates (100 mm x 100 mm) were placed onto a custom made supporting structure individually. The distance of the Bunsen burner to the steel substrate was set to 7 cm, so that the temperature on the surface of the steel substrate (with the aid of thermocouple) was at ~1000°C, imitating the environment similar to jet fire.

A thermocouple was attached to both front side and backside of the steel substrate each to examine the thermal insulation performance of the char formation. Data was retrieved from the data logger connected to the thermocouple and recorded every 1 minute.

As shown in Figure 3.7, the steel substrate was directly fired from the Bunsen burner

in perpendicular direction for 50 minutes. The steel substrate and char was then allowed to cool down naturally in room temperature.

Figure 3.7: Direct fire test setup.

3.2.3 Char Strength Test

The test procedure consists of measuring the amount of force required to crush the cooled char sample obtained from furnace fire for a certain penetration depth or height. Weights applied are increased with an increment of 50 g using individual mass pieces until char is totally crushed. The corresponding height of the char was measured with a steel rule (Figure 3.8).

3.3 Material Characterization

3.3.1 Qualitative and Quantitative Observations

Cooled down char samples from furnace fire test and direct fire test were inspected for their structural properties qualitatively, and the respective weight loss and height expansion for each samples were measured and calculated.

3.3.2 Thermogravimetric Analysis (TGA)

Cured intumescent coating was examined using TGA to determine its degradation

temperature and weight loss profile over temperature. The specimens were heated from 30^{o}C to 800^{o}C with a heating rate of 20^{o}C per minute.

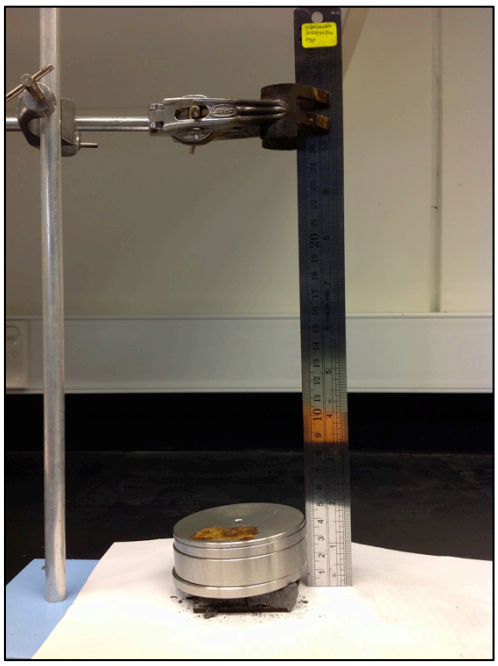


Figure 3.8 Char strength test setup.

3.3.3 X-Ray Diffraction Analysis (XRD)

XRD test was carried out on the char samples obtained from furnace fire test to investigate its crystal structure and chemical composition.

CHAPTER 4
RESULTS & DISCUSSION

4.1 Characterization of Intumescent Coating

The final weight and the dry film thickness (DFT) of each cured intumescent coating samples were measured and calculated for its overall volume and density. The average values were taken from 3 sets of samples, each consisting of 4 different formulations, i.e. RA1, RA2, RA3 and RA4. The detailed calculation of weight, thickness, volume and density can be found in Appendix A. The average thickness of the coating was 3.2 mm, with a range of +/- 20% difference due to manual application. The average coating density was found to be 1.11 g/cm^3. Thus, this epoxy based intumescent coating was considered as a low density and thick film coating. These properties were found to be closely similar with commercial intumescent coatings such as Chartek 7 [37].

4.2 Furnace Fire Test

4.2.1 Visual and Qualitative Observations

The side view, cross sectional view, and top view of the char samples are shown in Table 4.1. Qualitative observations of the char samples from the furnace fire test are shown in Table 4.2. Observations showed that the structural properties of the coatings were correlated to the length of fibres in the coating. Formulation with longer fibre length contributed to a more compact char structure with smaller pores, thus behave to be less crispy. This observation can be related to the degree of cross-linked network formed by the fibres within the char [24]. Although all charred samples lost their adhesion to the steel plate after slight movement, RA4 with fibre

length of 10 mm was found to be slightly more adhesive compared to the others. The one sided inclination of all samples may indicate a slightly non-uniform heat distribution inside the furnace. However the samples are still well exposed to heat from all directions in the enclosed space and therefore degradation can be considered to be complete.

Table 4.1: Visual observations of char samples from furnace fire test.

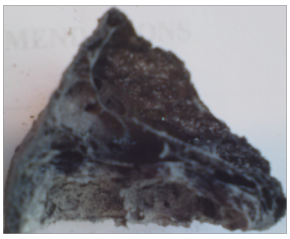
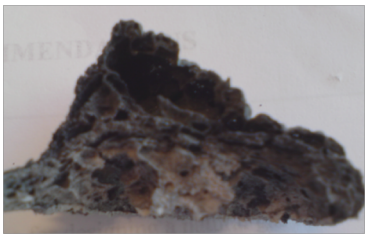
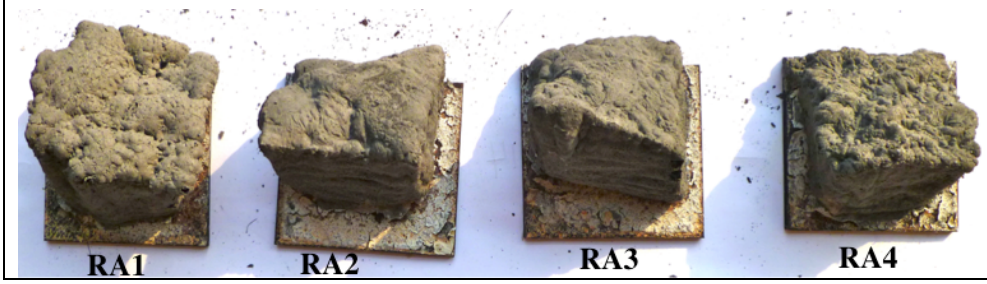
Sample	Side View	Cross Sectional View
RA1		
RA2		
RA3		
RA4		
Top View 		

Table 4.2: Qualitative observations of char samples from furnace fire test.

Description	Observation
Shape	Mountain-like, inclined towards the side
Surface Texture	Rough
Structure	Solid
Colour	Light Grey
Crispness	RA1: Most crispy; RA4: Least crispy
Porosity	RA1: Larger pores; RA4: Smaller pores
Compactness	RA1: Less compact; RA4: More compact
Adhesiveness to steel plate	RA1: Less adhesive; RA4: More adhesive

4.2.2 Weight Loss and Height Expansion

Table 4.3 and Figure 4.1 represent the average weight loss and height expansion in percentage for each formulation. The detailed calculations can be found in Appendix A. It is observed that as the fibre length in the intumescent coating increases, both weight loss percentage and height expansion percentage decreases. The findings showed that the increase in fibre length led to better thermal stabilization of the materials during char forming, which can be considered to be a benefit since a denser foam will provide more fire protection [2,6]. This finding also agreed with the observation of a more compact char structure with smaller pores.

Table 4.3: Average % weight loss and average % height expansion of each intumescent coating formulation from furnace fire test.

Formulation	Average % Weight Loss	Average % Height Expansion
R1	79.3 %	1071.4 %
R2	77.6 %	906.8 %
R3	74.3 %	772.0 %
R4	72.9 %	638.3 %

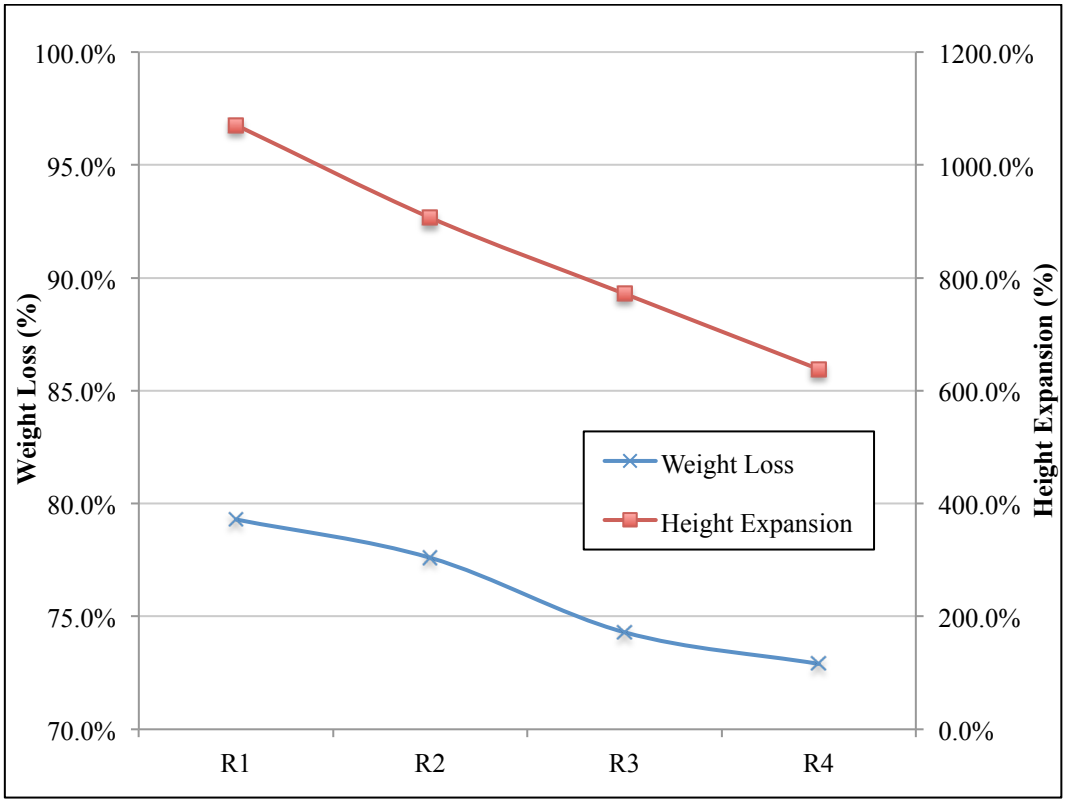


Figure 4.1: Weight loss and height expansion of each intumescent coating formulations obtained from furnace fire test.

4.3 Direct Fire Test

4.3.1 Visual and Qualitative Observations

Qualitative observations of the char samples from the direct fire test are shown in Table 4.4. The side view and top view of the char samples are shown in Figure 4.2 and Table 4.5 respectively. In this test, an additional controlled formulation (RR) without the inclusion of fibre reinforcements has been included for comparison purpose. Besides the temperature difference, the other difference of this test with the furnace fire test is the opportunity to observe the reactions of the samples over time as the test was done in an open environment. During the early stage of fire, strongest flame was observed for the controlled copy. RB1 was also observed to produce a strong flame yet comparatively lesser and reduced faster than the controlled formulation. The observations were similar for the remaining samples, with decreasing flammability for the samples with longer fibres. It was most reasonable to conclude that the flammability of the intumescent coating is related to the pyrolysis

of the material. With longer fibres, the degradation degree was smaller as the fibres form a more steady formation with the constituent resin, and thus allowed a better-structured char to be formed. Besides that, the coating with longer fibres appeared to retain its original form and structure, and adhered to the steel substrate better, as seen in Table 4.5. The controlled formulation with no fibre included was observed to be totally contracted and lost its adhesion to the steel substrate at the first few minutes of fire exposure. This finding further proved that longer fibres formed better crosslink structures during degradation, retaining the structure of the char.

Although the temperature of the direct fire (~1022^{o}C) was higher than the furnace fire (800^{o}C), the height expansion of coating was significantly lesser due to single direction and small concentrated area of the directed fire, and thus may not truly represented the behaviour of coating in case of real jet fire. Degradation may not be totally complete in the intumescent coating as the general colour of the char was charcoal black with a slightly grey area at the centre of the direct fire, as compared with the overall light grey coloured char from the furnace fire test.

Table 4.4: Qualitative observations of char samples after direct fire test.

Description	**Observation**
Flammability	RR: Strongest flame, flame reduced in 4-5 minutes; RB1: Strong flame, comparatively lesser than RR, flame reduced in 3-4 minutes; RB4: Smallest flame, flame reduced in 1-2 minutes
Height Expansion	Expanded most on the centre of flame, less expansion as compared to furnace fire test
Surface Texture	Rough
Structure	RR: Contracted, unable to maintain original form and structure after few minutes of fire exposure; RB1-4: More stable as compared to RR
Colour	Generally charcoal black; char around flame centre turns to light grey after long period of burning
Adhesiveness to steel plate	RR: Least adhesive; RB1: Less adhesive; RB4: More adhesive

Table 4.5: Visual observations of char samples after direct fire test.

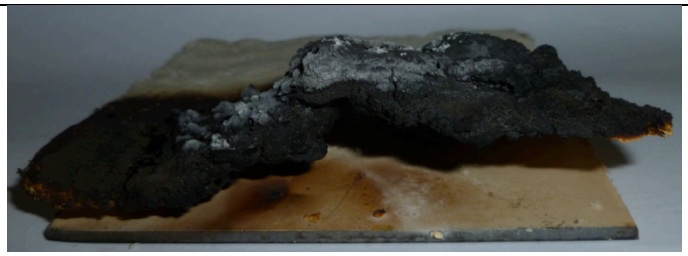
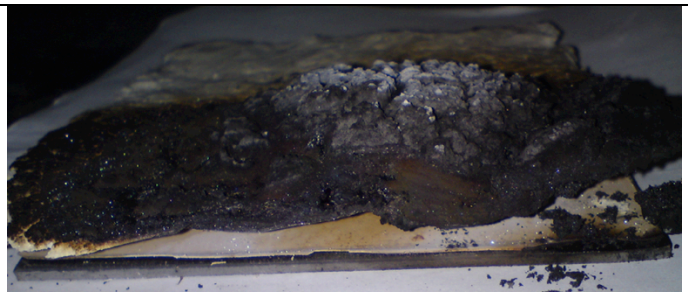
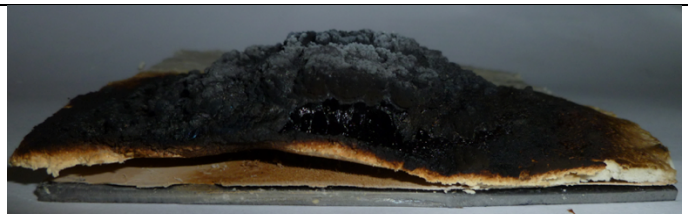
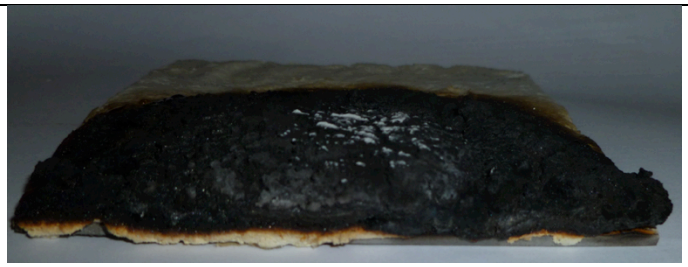
Sample	Side View
RR	
RB1	
RB2	
RB3	
RB4	

Figure 4.2: Top view of a char sample after direct fire test.

4.3.2 Thermal Insulation Performance

The temperature profiles measured at the backside of the intumescent-coated steel substrates for each formulation are reported in Figure 4.3. The temperature profile for an uncoated steel substrate was also included in the graph for comparison purpose. The average temperature of the direct fire, which is at the front side of the steel substrate, was approximately 1022^{o}C. As shown in Table 4.6, the average temperature at the backside of the intumescent-coated steel substrates for each formulation was taken after constant temperature was achieved, and the time to achieve the constant temperature was recorded. Generally, all intumescent-coated steel provided very good thermal insulation with a protection up to 85-88% of the original heat (400^{o}C) that reached the backside of the steel substrate if it was uncoated. Slight difference can be noted among the formulations, where the samples with longer fibres were able to achieve lower maximum temperature in a longer period. This finding shoed that degradation was slower and insulation was better for samples with longer fibres.

Table 4.6: Average constant temperature, T_{const}, at the backside of the coated steel substrates for each formulation and the time to achieve T_{const}.

Formulation	Average Constant Temperature, T_{const} (^{o}C)	Time to achieve T_{const} (min)
R1	72	8
R2	68	9
R3	66	10
R4	61	12

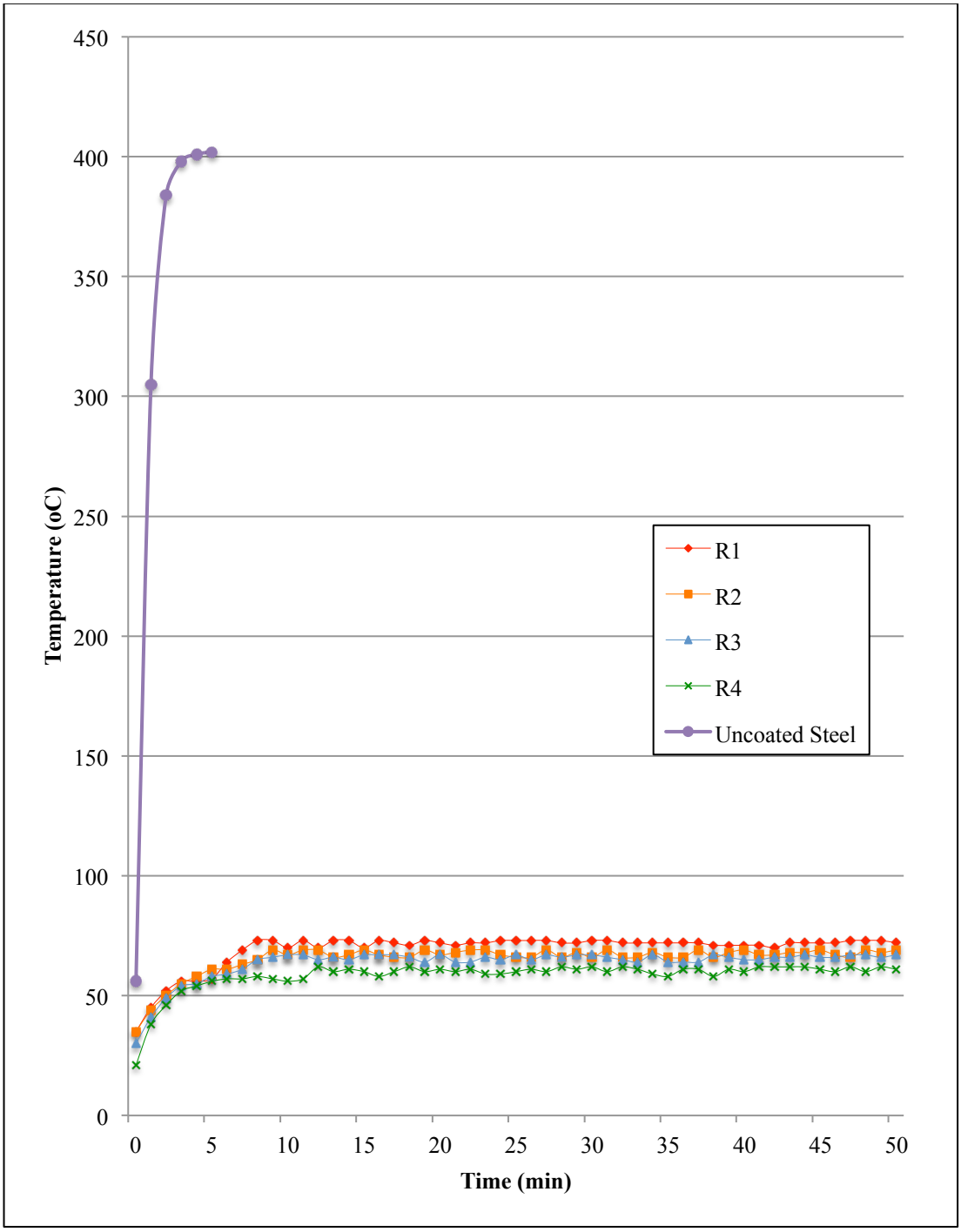


Figure 4.3: Temperature profile at the backside of the steel substrates as obtained from direct fire test.

4.4 Char Strength Test

Figure 4.4 shows a completely crushed char sample. From Figure 4.5, the char lost its strength to sustain its major structure at around 350 – 500 g of applied weight as indicated by the steep slope, with formulation R1 being the weakest. This finding concluded that the formulation with longer fibre length have a stronger char structure, thus provided better fire protection. Meanwhile, the residual height increased respectively with increasing fibre length in each formulation as shown in Figure 4.6. This is due to the increasing residual weight, which was observed and discussed earlier in Section 4.2.2.

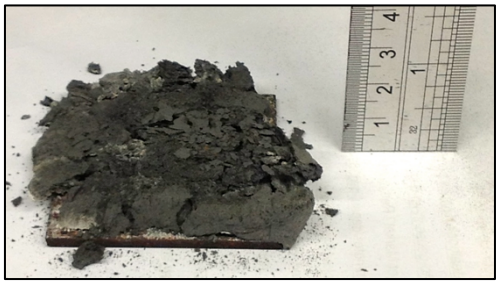


Figure 4.4: A crushed sample from char strength test.

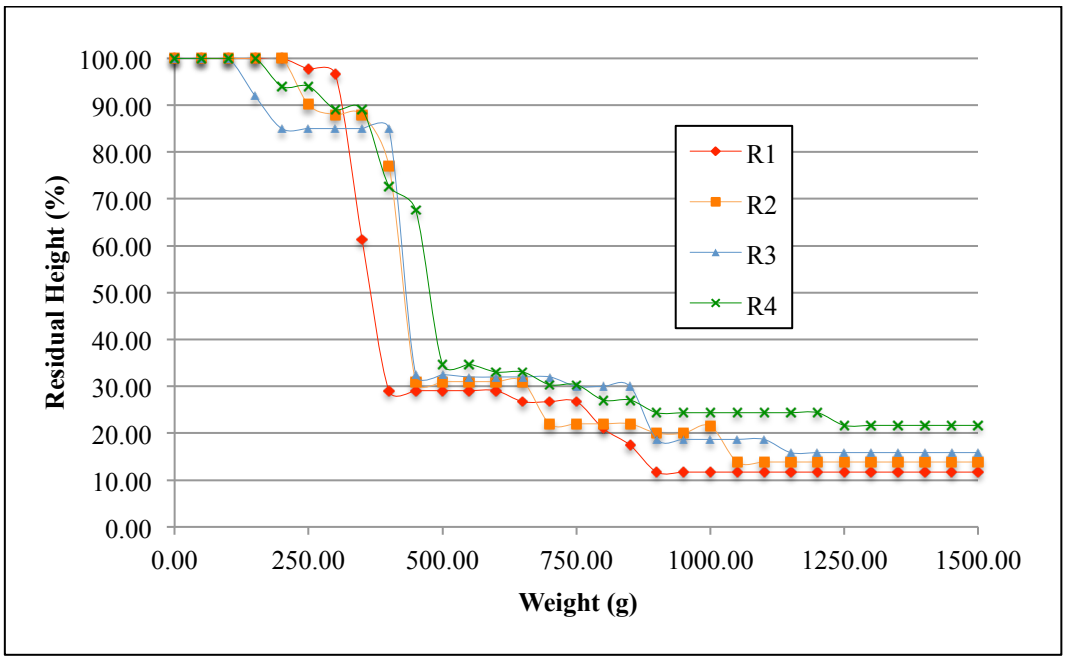


Figure 4.5: Graph of residual height (%) versus weight (g) applied onto char samples of each intumescent coating formulation.

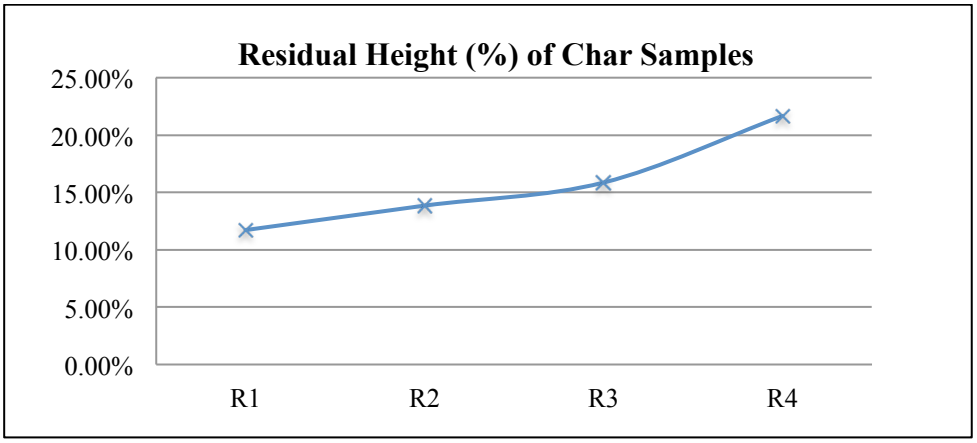


Figure 4.6: Residual height (%) of char samples for each intumescent coating formulation.

4.5 Thermogravimetric Analysis

Figure 4.7 represents the residual weight percent curve over increasing temperature for formulation R1 obtained from TGA. The curves for all formulations can be found in Appendix C. The degradation temperature T_d is found to be at ~380^{o}C, as indicated by the starting of the sharp slope in the plot. Similar results were observed for all four formulations and thus suggested that varying fibre lengths may not have an influence in the degradation temperature of the fibre reinforced intumescent coating. The degradation temperature is most likely depended only on the constituent resin, as reported elsewhere [2].

The formulation with longer fibres has more residual weight or less weight loss (Table 4.7), which agrees with the finding earlier in Section 4.2.2. However, the weight loss profile (Figure 4.8) obtained from TGA are generally lower from the furnace fire test by 10-15%, indicating the possibility of error during sample handling after furnace fire test.

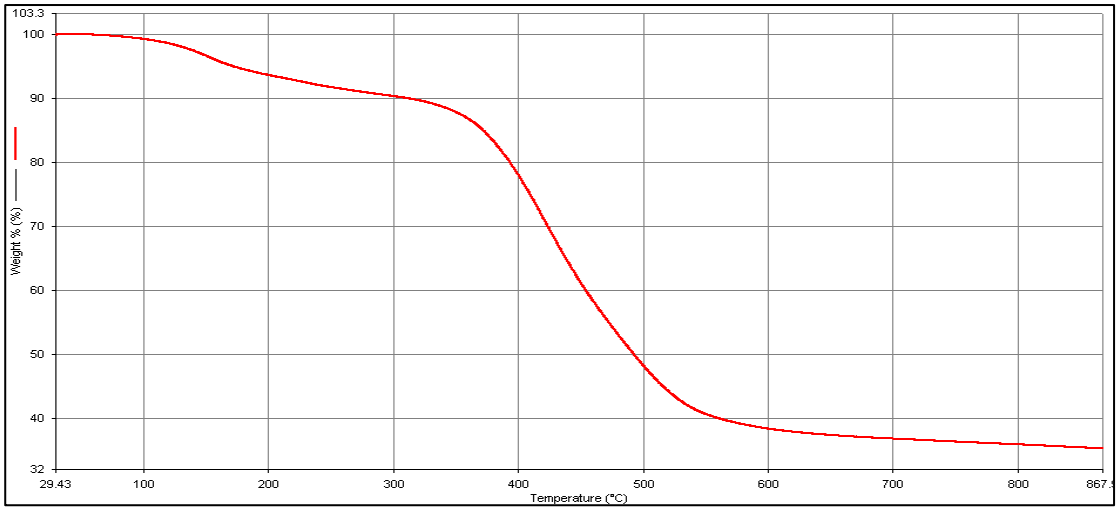


Figure 4.7: Graph of residual weight (%) versus temperature (^{o}C) for formulation R1.

Table 4.7: Residual weight and respective weight loss of each intumescent coating formulation at 800^{o}C.

Formulation	Residual weight (%)	% Weight Loss
R1	35.2 %	64.8 %
R2	36.3 %	63.7 %
R3	37.9 %	62.1 %
R4	38.6 %	61.4 %

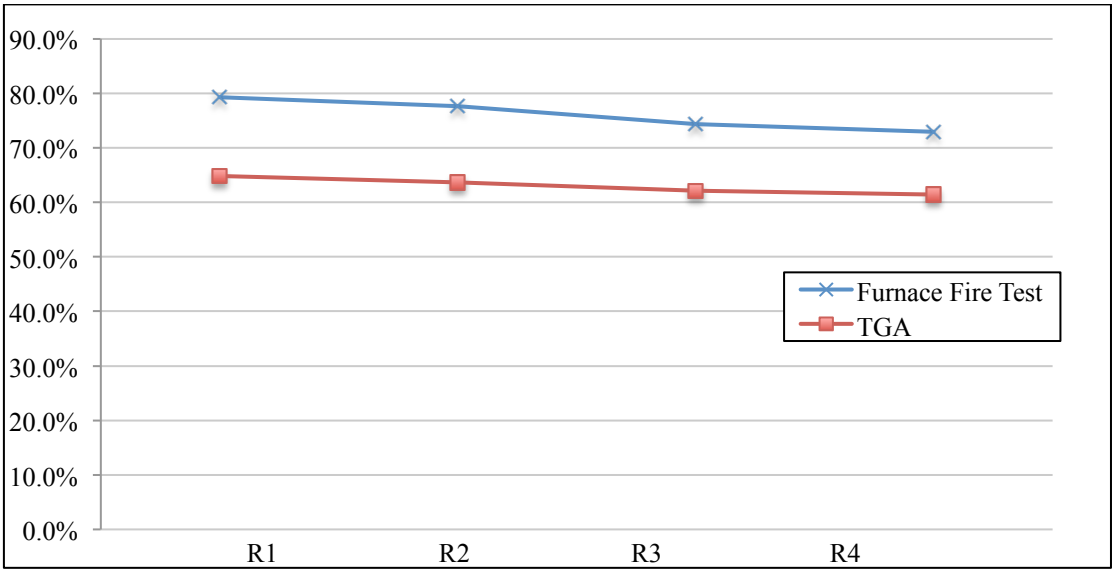


Figure 4.8: Weight loss profile of intumescent coating heated up to 800^{o}C as obtained from furnace fire test & TGA.

4.6 X-Ray Diffraction Analysis

Obvious noise was observed in the XRD curves, Figure 4.9, for all formulations, indicating that the chars are in amorphous state. After refining as in Figure 4.10, major peak for all four formulations was determined to be at 2θ = 24.5° pertaining to boron phosphate (BPO_4). Boron phosphate was known to impart great fire retardant properties. The next major peak of 2θ = 28° was equated to Sassolite or boric acid, H_3BO_3. Boric acid was proven to provide adhesion and good mechanical resistance properties. Boric acid was formed during dehydration to facilitate the formation of B_2O_3, which increased the viscosity and trap the gaseous decomposition products within the char causing it to expand [34]. This supported the result of high expansion and strong char produced as boric acid reacted positively with char-forming Rockwool fibre in the formulation. All four formulations of R1, R2, R3, and R4 displayed similar XRD patterns, indicating that varying lengths of Rockwool fibres do not influence the crystallography and chemical compositions of the intumescent coating.

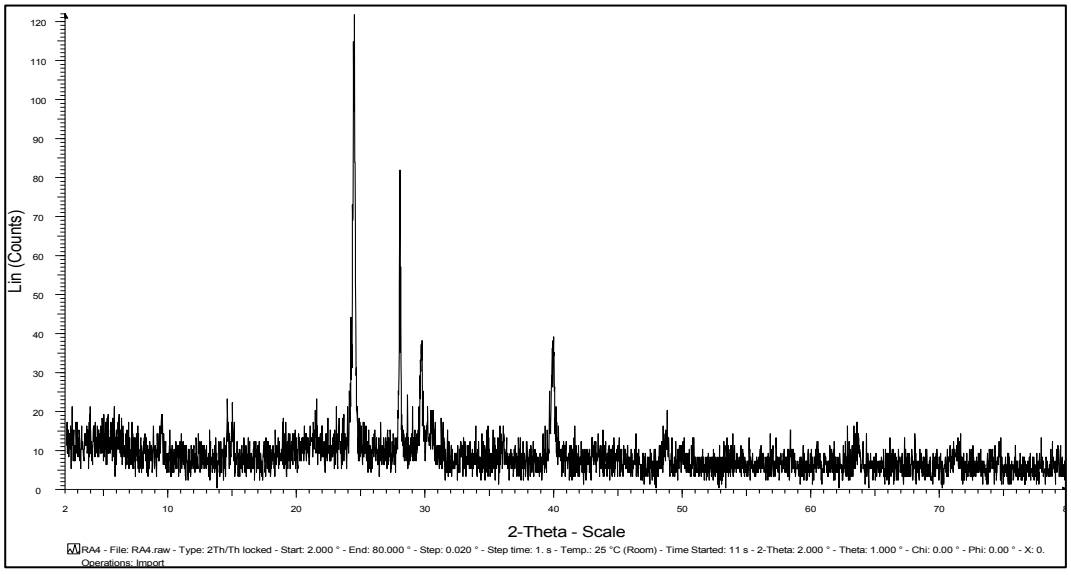


Figure 4.9: Raw XRD curve of char samples heated up to 800^{o}C from furnace fire test.

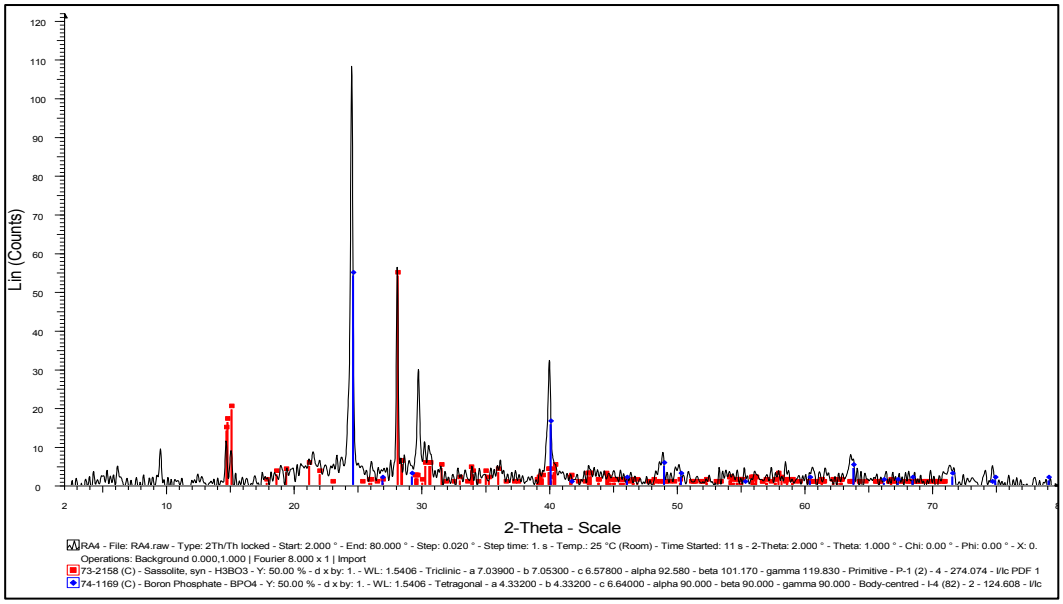


Figure 4.10: Refined XRD curve with major peaks matched.

CHAPTER 5
CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

Four formulations of intumescent coating reinforced with different Rockwool fibre lengths, i.e. 4 mm, 6 mm, 8 mm and 10 mm, and a controlled formulation, were developed and tested accordingly. In furnace fire test, the samples were heated up to 800^{o}C. Char expansion was measured between 6 to 10 times, while weight loss was between 72-79%. The formulation with the shortest fibres expanded and lost weight the most, and vice versa. In direct fire test, the samples were heated up to ~1022^{o}C. The findings showed that fibre reinforcement retarded flammability, greatly enhanced the adhesion of the coating to the steel substrate and the ability to retain its original form, and provided thermal insulation up to 85-88% of directed heat. The properties were further improved with increasing the Rockwool fibre lengths in the formulation. Char strength test was done on samples from furnace fire test and was found that the formulation with the longest fibres has the highest strength to sustain the char structure before destruction.

From the thermogravimetric analysis, degradation temperature was determined to be ~380^{o}C and was not influenced by the varying length of the Rockwool fibres, indicating dependence only on the constituent resin. Only the residual weight of samples was influenced and the observations were similar with the results found during furnace fire test. In the XRD analysis, varying the fibre lengths also did not influence the crystallography and chemical compositions of the intumescent char. All samples appeared to be in amorphous state and the major components determined were boron phosphate and boric acid.

5.2 Recommendations

Enhancement in the experimental investigation techniques can be done in the future using cone calorimeter to control the heating rate and provide better analysis of the intumescent mechanism. The thickness of the coating may be further reduced if application is automated using spray coating or spin coating. However the rheology of the coating may need to be further investigated as it is found to be highly viscous. Reduce in viscosity of the coating could also allow a wider range of fibre lengths to be used in the studies. The concept of rheology could also be used in the char strength test to find out the corresponding force required to penetrate a certain char depth with increasing heating temperature.

Since the experiments done in this study were mostly carried out manually, a possibility of human error could occur and deviate the actual results. Thus it is recommended that both manual and machine-aided experiments can be carried out in the future to further enhance the reliability of the results.

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APPENDIX A

Calculation of weight, thickness, volume and density of cured intumescent coating

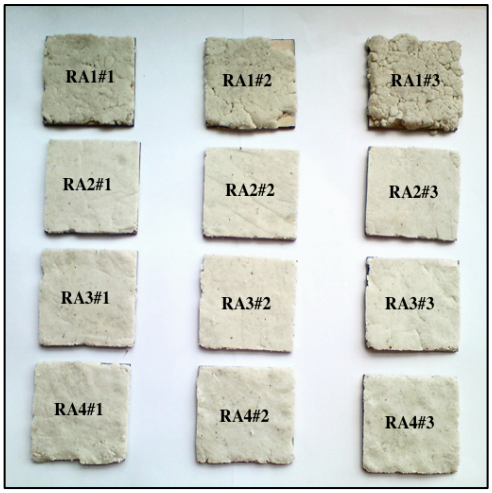

Figure A-1: Trimmed and cured intumescent coating samples.

Table A-1: Weight of cured coating for each sample.

Sample Weight	**RA1**			**RA2**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Primer Coated Steel (g)	49.29	51.25	49.38	51.25	49.89	49.56
Primer Coated Steel + Cured Coating (g)	58.98	58.40	65.44	59.22	58.68	57.75
Cured Coating (g) (Calculated Values)	**9.69**	**7.15**	**16.06**	**7.98**	**8.79**	**8.19**
Sample Weight	**RA3**			**RA4**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Primer Coated Steel (g)	50.45	51.43	50.39	50.35	49.43	49.36
Primer Coated Steel + Cured Coating (g)	58.59	58.06	58.59	58.84	57.42	57.45
Cured Coating (g) (Calculated Values)	**8.14**	**6.63**	**8.20**	**8.49**	**7.99**	**8.09**

Table A-2: Dry film thickness of cured coating for each sample.

Sample Thickness	**RA1**			**RA2**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Uncoated Steel (mm)	2.5			2.5		
Primer Coated Steel (mm)	2.7	2.7	2.7	2.7	2.7	2.7
Primer Coated Steel + Cured Coating (mm)	5.9	6.2	8.0	6.2	5.4	5.5
Coating DFT (mm) (Calculated values)	**3.2**	**3.5**	**5.3**	**3.5**	**2.7**	**2.8**
Sample Thickness	**RA3**			**RA4**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Uncoated Steel (mm)	2.5			2.5		
Primer Coated Steel (mm)	2.7	2.7	2.7	2.7	2.7	2.7
Primer Coated Steel + Cured Coating (mm)	5.5	5.3	6.4	5.5	5.4	5.5
Coating DFT (mm) (Calculated values)	**2.8**	**2.6**	**3.7**	**2.8**	**2.7**	**2.8**

Equation A-1:

Coating Volume (mm^3) = Coating DFT (mm) x Area of Steel Sheets (mm^2)

Area of Steel Sheets = 50 mm x 50 mm = 2,500 mm^2

Table A-3: Coating volume of the samples.

Coating Volume (mm^3)					
RA1			**RA2**		
#1	**#2**	**#3**	**#1**	**#2**	**#3**
8000.0	8750.0	13125.0	8812.5	6750.0	6937.5
RA3			**RA4**		
#1	**#2**	**#3**	**#1**	**#2**	**#3**
7000.0	6500.0	9187.5	6875.0	6812.5	6875.0

Equation A-2:

Coating Density (g/cm^3) = (1 mm^3/ 1000 cm^3) x Coating Weight (g) ÷ Coating Volume (mm^3)

Table A-4: Coating density of the samples.

Coating Density (g/cm^3)					
RA1			**RA2**		
#1	**#2**	**#3**	**#1**	**#2**	**#3**
1.21	0.82	1.22	0.91	1.30	1.18
RA3			**RA4**		
#1	**#2**	**#3**	**#1**	**#2**	**#3**
1.16	1.02	0.90	1.23	1.17	1.18

Average coating density

= (1.21 + 0.82 + 1.22 + 0.91 + 1.30 + 1.18 + 1.16 + 1.02 + 0.90 + 1.23 + 1.17 + 1.18) / 12

= **1.11 g/cm^3**

APPENDIX B

Calculation of weight loss and height expansion of char

Equation B-1:

% Weight Loss = [(Weight of Cured Coating – Weight of Char) ÷ Weight of Cured Coating] x 100%

Table B-1: Char weight and % weight loss for each sample.

Sample Weight	**RA1**			**RA2**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Char + Steel Sheet (g)	50.47	53.02	53.41	52.53	52.18	51.62
Steel Sheet (g)	49.29	51.25	49.38	51.25	49.89	49.56
Char (g)	**1.18**	**1.77**	**4.03**	**1.28**	**2.29**	**2.06**
Cured Coating (g)	9.69	7.15	16.06	7.98	8.79	8.19
% Weight Loss	**87.8**	**75.2**	**74.9**	**84.0**	**73.9**	**74.8**
Average % Weight Loss	(87.8 + 75.2 + 74.9) / 3 **= 79.3**			(84.0 + 73.9 + 74.8) / 3 **= 77.6**		
Sample Weight	**RA3**			**RA4**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Char + Steel Sheet (g)	52.52	53.19	52.46	52.58	51.59	51.63
Steel Sheet (g)	50.45	51.43	50.39	50.35	49.43	49.36
Char (g)	**2.07**	**1.76**	**2.07**	**2.23**	**2.16**	**2.27**
Cured Coating (g)	8.14	6.63	8.20	8.49	7.99	8.09
% Weight Loss	**74.6**	**73.5**	**74.8**	**73.7**	**73.0**	**71.9**
Average % Weight Loss	(74.6 + 73.5 + 74.8) / 3 **= 74.3**			(73.7 + 73.0 + 71.9) / 3 **= 72.9**		

Equation B-2:

% Change in height = [|DFT of Cured Coating – Thickness of Char| ÷ DFT of Cured Coating] x 100%

Table B-2: Char thickness and % height expansion for each sample.

Sample Thickness	**RA1**			**RA2**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Char + Steel Sheet Thickness (mm)	49.0	43.5	47.0	40.5	34.0	24.0
Steel Sheet Thickness* (mm)	2.5					
Char Thickness	**46.5**	**41.0**	**44.5**	**38.0**	**31.5**	**21.5**
Coating DFT (mm)	3.2	3.5	5.3	3.5	2.7	2.8
% Height Expansion	**1353.1**	**1071.4**	**739.6**	**985.7**	**1066.7**	**667.9**
Average % Height Expansion	(1353.1 + 1071.4 + 739.6)/3 = **1071.4**			(985.7 + 1066.7 + 667.9) / 3 = **906.8**		
Sample Thickness	**RA3**			**RA4**		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**
Char + Steel Sheet Thickness (mm)	**30.4**	**29.5**	24.0	21.0	30.0	17.5
Steel Sheet Thickness*	2.5					
Char Thickness	**27.9**	**27.0**	**21.5**	**18.5**	**27.5**	**15.0**
Coating DFT (mm)	2.8	2.6	3.7	2.8	2.7	2.8
% Change in height	**896.4**	**938.5**	**481.1**	**560.7**	**918.5**	**435.7**
Average % Height Expansion	(896.4 + 938.5 + 481.1) / 3 = **772.0**			(560.7 + 918.5 + 435.7) / 3 = **638.3**		

**Note: Assuming steel sheet thickness does not change after fire test.*

APPENDIX C

TGA curves of weight profile over temperature

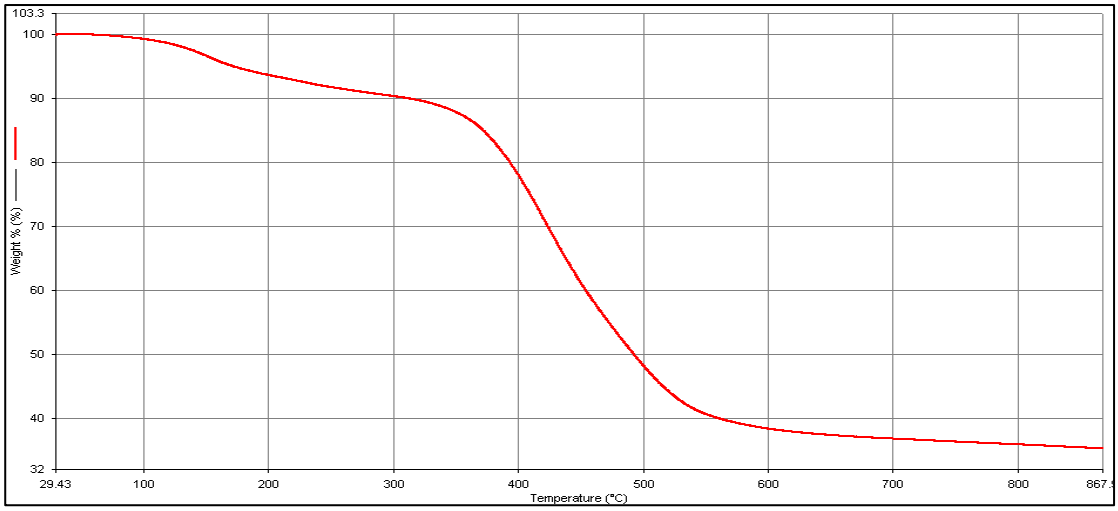

Figure C-1: Graph of residual weight (%) versus temperature (^{o}C) for formulation R1.

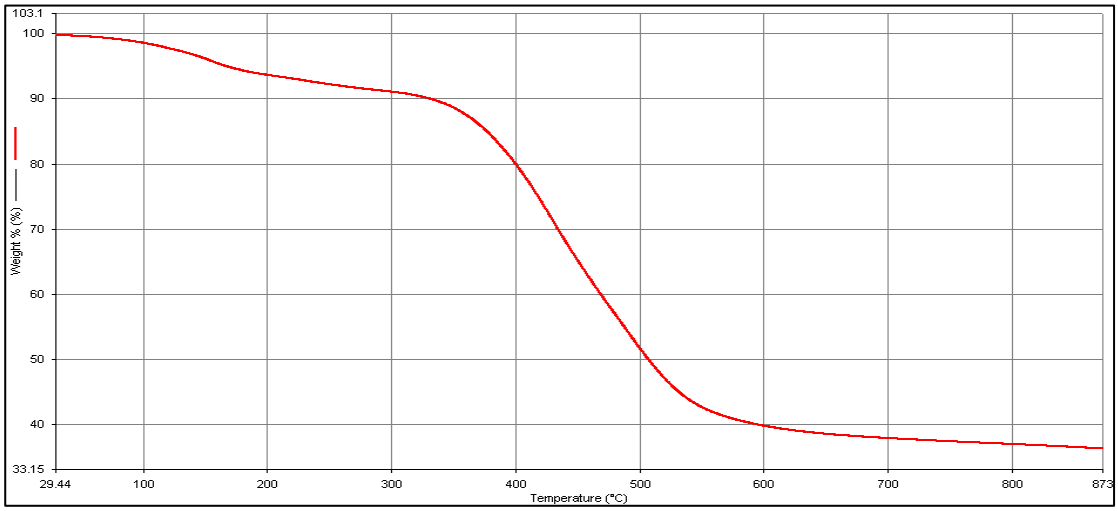

Figure C-2: Graph of residual weight (%) versus temperature (^{o}C) for formulation R2.

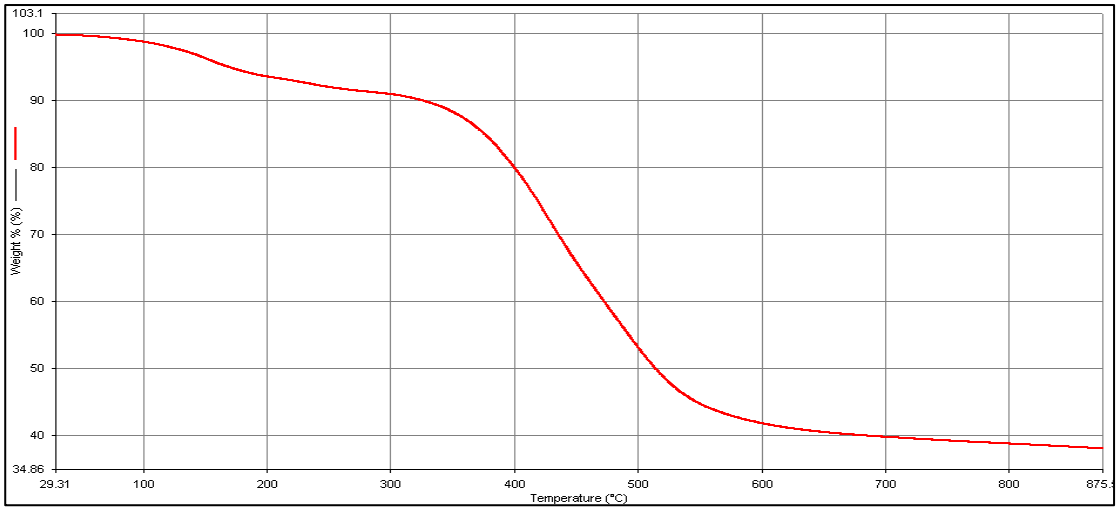

Figure C-3: Graph of residual weight (%) versus temperature (^{o}C) for formulation R3.

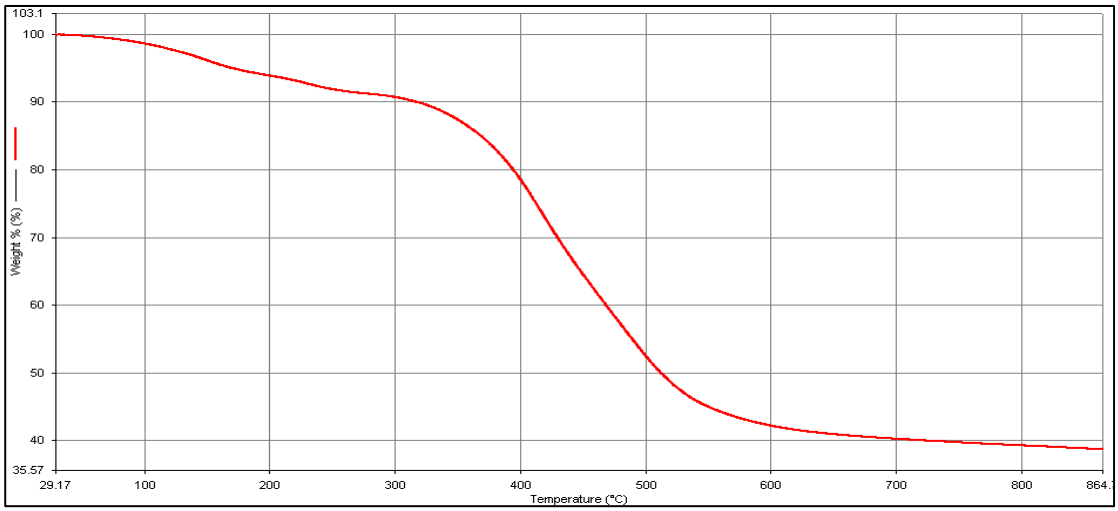

Figure C-4: Graph of residual weight (%) versus temperature (^{o}C) for formulation R4.